Flash Point Determination of Chemical Warfare Agents (CWAs) and Related Compounds

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The Importance of Flash Point

Flash point is the lowest temperature at which the vapor above the liquid specimen ignites, when an ignition source (test flame) is applied. Although this is an empirical measurement that will vary with method and test parameters, it is an important property which is used to assess the overall flammability hazard of a material and is a critical factor for determining agent stability during dissemination, safe storage and handling.

Different Methods Used to Determine Flash Point

- Closed Cup vs Open Cup
  - Closed cup - contained saturated vapor
  - Open cup - diluted vapor
- Both provide useful information
- Closed cup generally produces lower values than open cup
- Closed cup typically preferred since it reflects a worse case situation

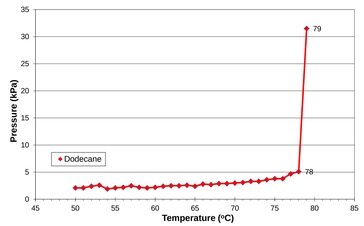
Different Types of Closed Cup Methods

- Small Scale a.k.a. Setaflash or Rapid Flash
  - Most widely used
  - Detects flash by visual observation
  - Sample size: 2-4mL per test temperature

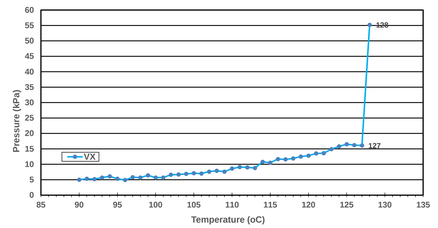
- Continuously Closed Cup
  - Preferred method for non-aqueous samples
  - Detects flash by a sharp, well-defined pressure increase
  - Minimizes sample size: 1mL per temperature range

Flash Point Results Using Continuously Closed Cup Method

- Typically provides stable pressure reading up to the point of the flash when testing flammable high purity single-phase samples

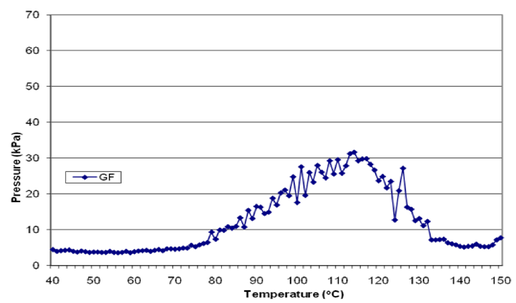


Pressure Profile of Dodecane



Pressure Profile of VX

- Gradual pressure increase followed by several pressure spikes provides inconclusive results which can be a sign of sample impurities and an indication of inappropriateness of the method.



Pressure Profile of GF

Concerns with Existing CWA Flash Point Data

Literature data is available on most CWAs and related compounds, however there has been some concern with the accuracy and method documentation involving several of these values, so new flash point measurements were conducted using closed cup methods.

- HD - (2,2'-dichloroethyl sulfide)
  - Currently accepted literature value is 105°C
  - Minimal information addressing sample purity, test method, and instrumentation
- CECPRS - (2-chloroethyl-3-chloropropyl sulfide)
  - Literature data not found
- VX – {O-ethyl-S-[2(diisopropylamino)ethyl] methylphosphonothiolate}
  - Currently accepted literature value is 159°C
  - McCutchan and Young micro method (open cup)
- VM – [O-ethyl S-(2-diethylaminoethyl) methylphosphonothiolate]
  - Currently accepted literature value is 236°C
  - McCutchan and Young micro method (open cup)
  - Inappropriateness of method due to sample loss
- GF – (cyclohexyl methylphosphonofluoridate)
  - Currently accepted literature value is 94°C
  - Calculated value

Summary of Flash Point Results for HD, CECPRS, VX, VM, and GF

Compound	Previous Flash Point Data (˚C) (Method) Year	Updated Flash Point Data (˚C)	
		(Closed Cup Method) Year	Purity
HD	105 (Method?) 1944	104 (CCCFP)[1] 2009	97.3%
		110 (Small Scale)[2] 2009	97.6%
CECPRS	Data not available	113 (CCCFP)[1] 2009	99%
VX	159 (Open Cup)[3] 1956	127 (CCCFP)[1] 1999	95.8%
VM	236 (Open Cup)[3] 1956	107 (CCCFP)[1] 2012	96%
GF	94 (Calculated)	119 (Small Scale)[2] 2011	>99%

[1] ASTM D6450 Flash Point by Continuously Closed Cup (CCCFP) Tester using Grabner FLPH Miniflash tester.
[2] ASTM D3828 Flash Point by Small Scale Closed Cup Tester using Koehler Rapid Flash Tester K16502.
[3] McCutchan and Young Open Cup Micromethod.

Approved for Public Release